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Elevated temperature stability of carbon-fibre, nickel-matrix composites: morphological and mechanical property degradation

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Carbon-fibre, nickel-matrix composites exhibit excellent microstructural stability when the composites are isothermally annealed at temperatures to 1200°C for several hundred hours; morphological degradation of the reinforcing phase takes place at temperatures in excess of 1250°C. The degradation temperature is less than 1200°C when the composites are subjected to thermal cycling or temperature gradients.

The mechanical properties of fibres extracted from annealed composites are approximately 40 to 50% less than the corresponding properties of unprocessed stress-graphitized fibres. Substantial mechanical property degradation occurs during the hot-forming of nickel-coated fibres into dense shapes. Subsequent re-annealing of the dense composite results in additional fibre damage. Most of this additional degradation occurs within the first few minutes from the start of the anneal; prolonged isothermal annealing does not result in further property degeneration.

1. Introduction

Fibre-reinforced metal composites are usually exposed to elevated temperatures in the course of fabrication and, often, during their use. At sufficiently high temperatures, appreciable mass transport, due to differences or gradients of the chemical potential, will take place even in ostensibly stable polyphase systems; any form of material transport which is accompanied by morphological and mechanical property degradation of the fibres is of signal importance because the mechanical properties of the composite ultimately depend upon physical property retention by the reinforcing phase.

Mass transport which results in fibre degradation is generally caused by one or more of several mechanisms. Foremost are chemical reactions between the matrix and the reinforcing phase which lead to the formation of new phases [1-3]; when limited, these interactions are often useful as a means of promoting strong matrix-fibre interfacial bonding [4].

Although catastrophic chemical reactions can be virtually eliminated by the judicious selection of the matrix and the reinforcing phase, significant degradation can occur even in those systems which are chemically inactive internally. Here, fibre instability is due chiefly to the effects of temperature, or of curvature (capillarity), on the chemical potential.

Because the chemical potential is temperature dependent, a gradient in temperature, or thermal cycling, can lead to substantial microstructural changes by a mechanism of solution at high temperatures followed by reprecipitation at lower temperatures; the matrix plays a direct role only as a medium for volume transfer of the reinforcing phase.

Capillarity can also induce severe microstructural changes; the effect of curvature on the chemical potential, described by the familiar Gibbs-Thomson equation [5], furnishes the driving force for the transport of matter, often over appreciable distances. There are three principal effects to consider: (i) if the shape of the internal (reinforcing) phase is not governed by external factors such as rapid changes in size, it will attempt to assume the equilibrium shape [5, 6]; in most cases the equilibrium shape will not be in the form of a long fibre; (ii) Ostwald ripening or coarsening of the internal phase [7–9]; and, (iii) concomitant segmentation and spheroidization of fibres due to the presence of surface undulations or other shape irregularities [10].

Item (iii), segmentation, is particularly important. This problem has been considered by Nichols and Mullins [10] by analysing the shape-preserving conditions for long rods slightly perturbed from a cylindrical geometry. The amplitude of a longitudinal perturbation (sinusoidal) will either grow or decay, depending upon the ratio of the wavelength to the cylinder diameter, as well as on the mechanism of material transport [10]; longitudinal undulations which grow result in segmentation of the long fibres. On the other hand, radial surface irregularities, such as flutes, are inherently unstable [10]. Several rate-controlling steps for capillarity-related material transport have been discussed by Li and Oriani [11].

In the case of carbon fibres, degradation by recrystallization can also occur [12–14]; it is well established that certain metals such as nickel act as catalytic agents for the graphitization of carbon at low temperatures (e.g., 1000° C).

This investigation was undertaken to examine the isothermal stability of carbon-fibre, nickel-matrix composites in the temperature range 750 to 1250° C; the effects of thermal cycling and thermal gradients were also examined.

Compatibility studies of nickel-coated single carbon fibres, as well as of nickel-coated bundles, have been conducted by other investigators [12–16]; those results suffered from the extraneous effects of a free nickel-vapour interface. The work reported here differs; composites, formed by hot-pressing nickel-coated carbon fibres into dense shapes, were examined for microstructural and physical property changes induced by subsequent heat-treatment.

2. Experimental

The carbon fibres used in this study were made from a cellulose II (rayon) precursor and had a nominal value of 50 million psi for the Young's modulus; marketed under the trade name "Thornel-50", these high-modulus fibres are produced from low-modulus carbon fibres by stretching the fibres at elevated temperatures [17]. This process, termed stress-graphitization, produces a fine structure of highly-oriented microfibrils by eliminating, in addition to internal porosity, the kinks and distortions within the ribbon-like graphite layer planes [17–19]; in essence, the stacks become more nearly perfect with increased stretching and heat-treatment temperature. However, the ordered structures imparted by stress-graphitization are not stable and undergo partial reversion or relaxation when the fibres are reheated without an externally applied stress at even moderate temperatures [19]; relaxation, in the temperature range 750 to 1200° C, is accompanied by a significant ($\sim$20%) decrease in the fibre mechanical properties [19].

An electrolytic deposition technique, described in detail by Sara [15], was used to coat each filament in the yarn; the desired fibre loading, 45 (nominal) vol % fibre, was obtained by controlling the thickness of the nickel coating. A Watt's plating solution was used.

The nickel-coated fibres were aligned in graphite moulds and were hot-pressed at a temperature of 1050° C, and under a vacuum of 10^{-3} torr, to form rectangular $\frac{1}{8} \times \frac{1}{16} \times 1$ in. test bars; complete densification was usually obtained within 1 h at a forming pressure of 2000 psi. The bulk density and Young's modulus of each hot-pressed test specimen was measured and those specimens which showed spurious values for either the density or the modulus were discarded; the Young's modulus was determined by using standard sonic methods. In addition to non-destructive testing, a number of specimens were examined for fibre damage and porosity; the characteristic tensile strength of the hot-formed composites was also determined. These physical properties are shown in Table I and are in good agreement with these reported by Sara [15] for composites fabricated and tested by using identical methods.

In Table I, the characteristic mechanical properties of the hot-pressed composites are compared with theoretical values calculated by using the rule-of-mixtures [20]; the calculations were made by using the nominal mechanical properties for: (a) as-received "Thornel-50"; (b) relaxed stress-graphitized fibres [19]; and (c) fibres extracted from the hot-pressed composites. The results indicate that mechanical property degradation of the reinforcing fibres occurs during hot-forming; this degradation cannot be attributed to fibre relaxation alone.

The hot-pressed bar specimens were subsequently packed in high-purity nickel powder and re-annealed for periods ranging from

TABLE I Mechanical properties of carbon-fibre, nickel-matrix composites; comparison of measured values with values calculated using the rule-of-mixtures.

	Measured	Theoretical*		
		(a)	(b)	(c)
Bulk density, g/cm^3	5.542 ± 0.114	—	—	—
vol% fibre loading	46.3	—	—	—
Young's modulus, 10^6 psi	32.8 ± 1.49	40.0	35.0	33.6
Fracture stress, 10^3 psi	79.5 ± 3.0	136.0	108.0	84.2

(a) As-received "Thornel-50": tensile strength, $\sigma_f = 28.4 \times 10^4$ psi; Young's modulus, $E_f = 49.3 \times 10^6$ psi [19].
(b) Relaxed stress-graphitized fibres: $\sigma_f = 22.0 \times 10^4$ psi; $E_f = 40.0 \times 10^3$ psi [19].
(c) Extracted fibres: $\sigma_f = 17.5 \times 10^4$ psi; $E_f = 38.0 \times 10^6$ (this work).

* In these calculations the Young's modulus and the flow stress of the nickel matrix were taken to be 30.0×10^6 psi and 10000 psi respectively [28].

0.5 to 200 h, at temperatures in the range 750 to 1250°C; the furnace chamber was evacuated to 10^{-5} torr prior to the annealing which was done in a flowing argon atmosphere. The temperatures were monitored with Pt/Pt-13% Rh thermocouples and temperature control was within ±2°C.

In addition to the isothermal anneals, specimens were subjected to either thermal cycling or thermal gradients for periods of up to 24 h; again, the atmosphere was flowing argon. Thermal cycling was done at a mean temperature of 1100°C, cycled within ±10° to ±50°C; the cycling was accomplished by missetting the proportional band adjustment of the temperature controller.

Controlled temperature gradients were obtained by mounting specimens onto a water-cooled copper probe and placing the probe into a constant temperature hot-zone. The hot end of the specimen was measured by means of a thermocouple and this temperature was maintained at the ambient temperature of the furnace, 1100° ± 2°C; the temperature of the hot-end as well as the temperature gradient was kept constant by adjusting the flow of water through the probe.

After annealing, the specimens were surface ground, tested, and the microstructure was examined. In addition, fibres were extracted from annealed composites by dissolving the nickel matrix in a hydrochloric acid solution. For mechanical property testing, individual fibres were mounted onto a cardboard backing with a 2 cm long window; the filament and backing were mounted between the grips of an Instron tester and the backing was severed prior to testing. The fibre cross-sectional areas were determined from photomicrographs by the method of point counting [21]. It was verified that the hydrochloric acid treatment did not alter the mechanical properties of the extracted fibres.

Student's t-test was used to evaluate the significance of the difference between the magnitudes of each mechanical property before and after annealing; this procedure distinguishes significant deviations from chance fluctuations which may arise because of random sampling variations alone. The levels of significance adopted in this study, together with the interpretation to be put on them, are those recommended by Davies [22]

3. Results and discussion

3.1. Morphological stability

The microstructures of carbon-fibre, nickel-matrix composites are generally stable and exhibit virtually no change when the composites are annealed for periods of up to 200 h at temperatures less than 1200°C; fibre degradation occurs only after prolonged isothermal annealing at temperatures approaching the nickel-carbon eutectic temperature, 1320°C [23]. For example, the microstructures of longitudinal and transverse sections of a composite annealed for 0.5 h at 1200°C, shown in Fig. 1, are very similar to the structure of the hot-pressed specimens prior to subsequent annealing. The only distinguishable changes seem to be decay of the normally fluted surface to a more nearly cylindrical geometry, and, bridging between adjacent carbon fibres. The amount of bridging increases with increased anneal temperature and time and is similar to neck growth commonly observed during the liquid phase sintering of finely subdivided powders [24]. The decay of the radially

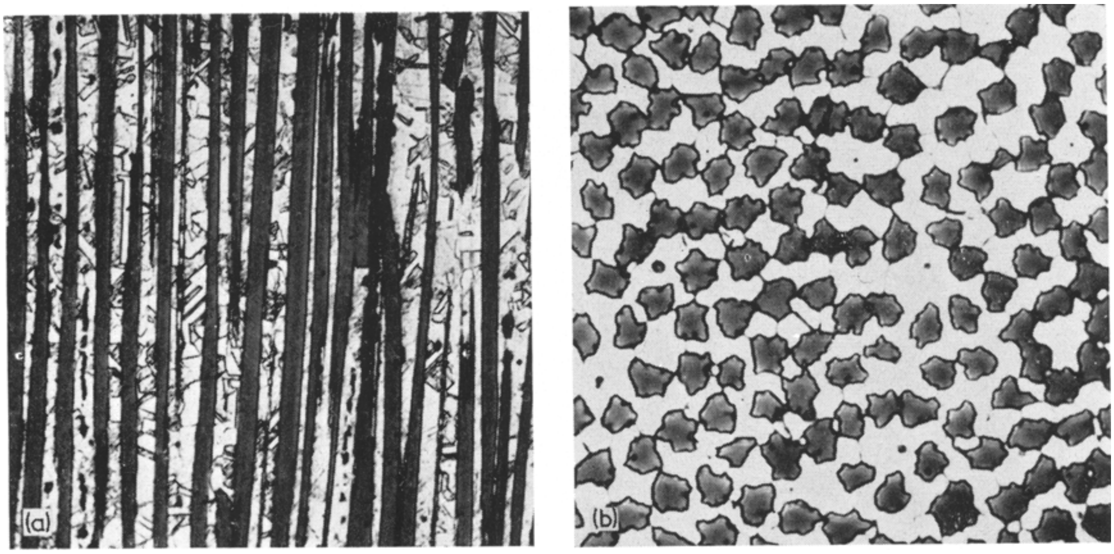

Figure 1 Microstructure of a carbon-fibre, nickel-matrix composite annealed isothermally for 0.5 h at 1200°C: (a) longitudinal section, (b) transverse section.

fluted surface is consistent with the Nichols-Mullins analysis discussed earlier [10]. Polished longitudinal sections were examined for optical anisotropy; the carbon fibres showed no evidence of additional graphitization as a result of isothermal annealing.

On the other hand, the microstructure of a composite annealed for 24 h at 1250°C, shown in Fig. 2, clearly shows massive degradation of the carbon fibres. The mechanism does not appear to be identical to that postulated by Nichols and Mullins [10]; that is, fibre degradation is not caused by segmentation. Rather, the fibres seem to recrystallize at temperatures in excess of 1200°C. The newly formed, nonfibrous internal phase particles are optically anisotropic and appear to be highly graphitic; in contrast, the optical anisotropy of fibres which

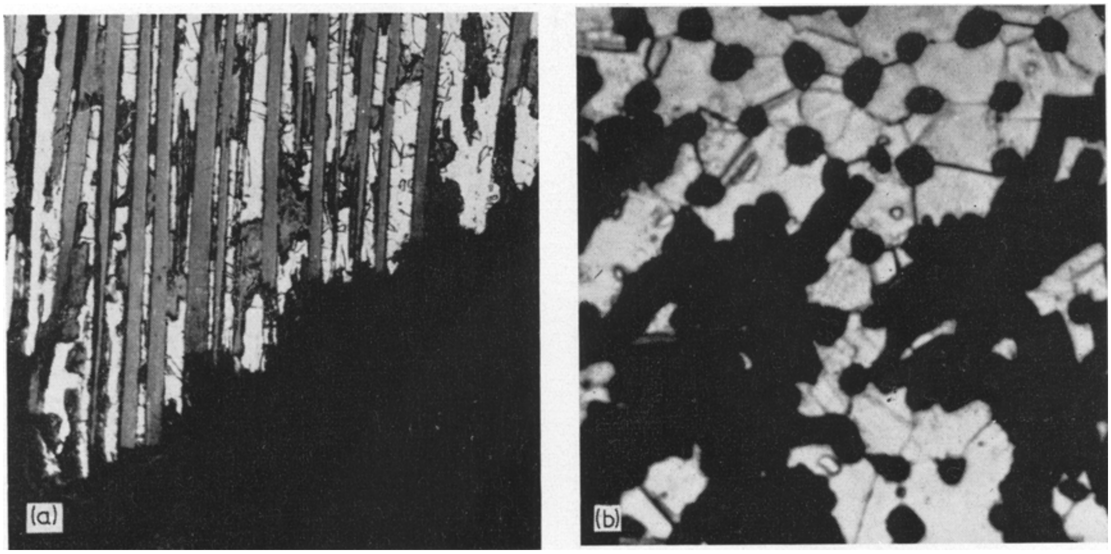

Figure 2 Microstructure of a carbon-fibre, nickel-matrix composite annealed isothermally for 24 h at 1250°C; the fracture surface of a tensile specimen is shown: (a) longitudinal section, (b) transverse section.

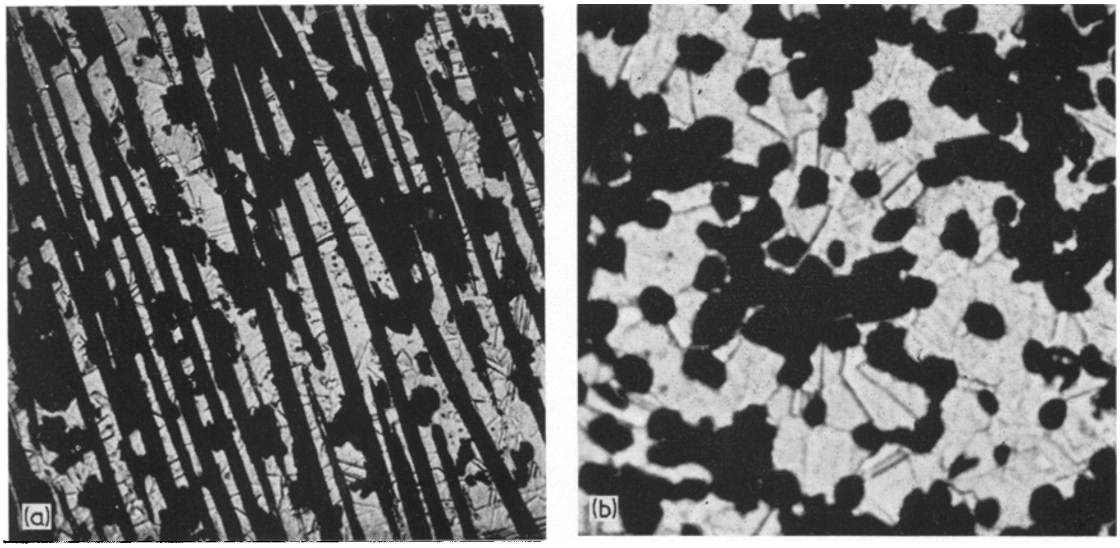

Figure 3 Microstructure of a carbon-fibre, nickel-matrix composite subjected to thermal cycling at a mean temperature of 1100° ± 2°C for 24 h: (a) longitudinal section, (b) transverse section.

are not morphologically affected by the anneal remains unchanged.

It was not possible to extract fibres from composites annealed at 1250°C because of fibre degradation; in all other cases, even for composites annealed for 200 h at 1200°C, fibre extractions were accomplished easily.

In contrast to the exceptional morphological stability of dense composites, loose bundles of nickel-coated carbon fibres degrade when they are heated isothermally to temperatures considerably lower than 1000°C; the instability of loose bundles, described in detail by other investigators [12–15], was verified in the present study.

The microstructural degradation which can be induced by thermal cycling is illustrated in Fig. 3; this structure was obtained by exposing a bar specimen to a mean temperature of 1100°C, cycled within ±10° to ±50°C, for a period of 24 h. A considerable volume fraction of the reinforcing phase has recrystallized as graphite.

Temperature gradients can also lead to severe microstructural changes; longitudinal microstructures located at different positions along a temperature gradient are shown in Fig. 4. The hot end of the bar specimen was maintained at 1100 ± 2°C for 24 h. Substantial volume transport of carbon along the gradient has resulted in the segmentation of the fibres. The original alignment of the long fibres is still apparent; the remaining segments, as well as the reprecipitated particles, are highly graphitic.

In summary, carbon-fibre, nickel-matrix composites are morphologically stable when heated isothermally at temperatures to 1200°C for extended periods of time. Composites heated to 1250°C undergo extensive microstructural changes; degradation at temperatures in excess of 1200°C is caused by recrystallization rather than segmentation or other capillarity-related phenomena. Fibre damage can be induced at temperatures lower than 1200°C by thermal cycling or by thermal gradients.

3.2. Mechanical property degradation

The bulk density of the annealed composites invariably decreased by several per cent; specifically, the bar specimens swelled slightly in the directions normal to the aligned fibres. Correspondingly, the fibres expanded radially by 10 to 15%; electron microprobe analysis of extracted fibres did not establish the presence of nickel within the fibre bulk volume and the magnitude of the increase in fibre diameter is about equal to that for stress-graphitized carbon fibres annealed in the absence of nickel [19]. Consequently, the radial expansion is probably due to fibre relaxation and is probably not caused by the diffusion of nickel into the fibres; the driving force is the release of residual strain energy induced into the fibres during the stress-graphi-

Figure 4 Microstructure of a carbon-fibre, nickel-matrix composite subjected to a temperature gradient for 24 h: (a) hot end (1100° C), (b) intermediate temperature region, and (c) cool end.

tization process [19, 25]. This radial expansion precluded analysis for Ostwald ripening of the reinforcing phase; however, metallographic examination of transverse sections showed that the number of fibres per unit area remained constant with annealing time, indicating that fibre coarsening does not occur to any appreciable extent even at temperatures of 1200° C. In contrast to this result, appreciable coarsening occurs in Al_3Ni whisker-reinforced aluminium [26].

The Young's modulus and fracture strength of the annealed composites, together wite the mechanical properties of extracted fibres, are given in Tables II and III. The null-hypothesis analysis indicates that there is no significant effect of either temperature or time on the mechanical properties. However, mechanical property degradation does occur, but within a time scale less than 30 min; the magnitude of this degradation is essentially the same for all temperatures in the range 750 to 1200° C.

This point is illustrated in Figs. 5 and 6. Here, the fracture strengths of composites, and of extracted carbon fibres, annealed at 925 and 1100° C, are plotted as a function of the anneal time; the standard error bars of the data points have been omitted for purposes of clarity. The data is similar for composites annealed at other temperatures in the range 950 to 1200° C. The effect of annealing time is clear; the tensile strength of the composite, and of the reinforcing fibres, initially decreases very rapidly, almost catastrophically; additional isothermal annealing results in virtually no additional loss in strength. The behaviour of the Young's modulus, shown in Figs. 7 and 8 for the 925 and 1100° C anneals, is similar. It is pertinent to note that, based upon the measured diffusion coefficients of nickel in pyrolytic graphite [27], 0.5 h is ample time for diffusion of nickel (or other impurities) through appreciable distances in the fibre; however, as noted earlier, the presence of

TABLE II Fracture stress, σ_c, and Young's modulus, E_c, of isothermally annealed composites

Anneal time (h)		Anneal temperature (°C)				
		750	925	1000	1100	1200
0.5	σ_c, 10^3 psi	67.4 ± 0.3	71.3 ± 3.2	57.8 ± 6.6	80.8 ± 0.1	69.1 ± 8.3
	E_c, 10^6 psi	33.4 ± 1.1	33.3 ± 0.1	33.3 ± 0.3	32.7 ± 0.2	32 7 ± 0.3
1	σ_c, 10^3 psi	72.3 ± 5.1	66.0 ± 1.8	70.7 ± 22.4	71.1 ± 1.6	68.1 ± 2.7
	E_c, 10^6 psi	33.3 ± 1.0	33.0 ± 0.7	33.7 ± 0.1	32.6 ± 0.5	33.3 ± 0.3
2	σ_c, 10^3 psi	77.0 ± 4.4	64.2 ± 10.0	73.3 ± 3.0	65.2 ± 2.4	75.3 ± 8.3
	E_c, 10^6 psi	32.7 ± 0.4	33.6 ± 0.4	33.1 ± 0.5	32.4 ± 0.1	32.5 ± 0.3
10	σ_c, 10^3 psi	64.0 ± 14.0	76.5 ± 9.6	72.0 ± 8.4	76.7 ± 7.1	58.4 ± 8.0
	E_c, 10^6 psi	32.1 ± 1.4	32.7 ± 1.7	33.4 ± 0.4	33.1 ± 0.3	33.5 ± 0.3
100	σ_c, 10^3 psi	59.6 ± 8.3	70.6 ± 1.4	60.1 ± 12.9	62.7 ± 3.6	68.0 ± 5.1
	E_c, 10^6 psi	30.9 ± 1.4	31.8 ± 1.0	32.9 ± 0.3	32.3 ± 0.3	31.1 ± 0.2
200	σ_c, 10^3 psi	60.4 ± 15.9	50.6 ± 7.6	64.0 ± 16.4	74.3 ± 3.7	62.3 ± 14.8
	E_c, 10^6 psi	33.3 ± 1.6	28.1 ± 0.9	32.7 ± 0.6	32.4 ± 0.3	31.3 ± 0.3

TABLE III Fracture stress, σ_f, and Young's modulus, E_f, of carbon fibres extracted from isothermally annealed composites

Anneal time (h)		Anneal temperature (°C)				
		750	925	1000	1100	1200
0.5	σ_f, 10^3 psi	144 ± 44	175 ± 45	152 ± 52	155 ± 52	126 ± 41
	E_f, 10^6 psi	34.5 ± 6.1	35.7 ± 5.3	25.6 ± 3.2	29.0 ± 8.3	20.2 ± 8.1
1	σ_f, 10^3 psi	180 ± 66	146 ± 46	169 ± 41	164 ± 45	137 ± 50
	E_f, 10^6 psi	35.0 ± 8.8	31.4 ± 8.0	30.8 ± 5.2	34.6 ± 5.8	35.4 ± 5.2
2	σ_f, 10^3 psi	126 ± 38	136 ± 49	132 ± 40	108 ± 62	134 ± 44
	E_f, 10^6 psi	23.6 ± 6.0	31.9 ± 9.2	29.8 ± 4.0	31 7 ± 10.7	33 4 ± 2.7
10	σ_f, 10^3 psi	142 ± 49	140 ± 40	145 ± 66	135 ± 54	98 ± 43
	E_f, 10^6 psi	37.2 ± 7.3	30.7 ± 8.8	30.0 ± 4 5	29.8 ± 7.2	28.3 ± 4.9
100	σ_f, 10^3 psi	156 ± 27	110 ± 41	209 ± 32	130 ± 40	122 ± 56
	E_f, 10^6 psi	34.2 ± 4.3	25.0 ± 8.7	43.0 ± 7.5	28.2 ± 7.9	25.6 ± 4.1
200	σ_f, 10^3 psi	171 ± 58	90 ± 40	83 ± 40	122 ± 42	110 ± 39
	E_f, 10^6 psi	34.6 ± 4.6	23.8 ± 4.7	25.2 ± 8.4	28.5 ± 6.7	18.4 ± 7.4

nickel was not firmly established by electron microprobe analysis of extracted fibres.

In principle, the fracture strength of a composite, $(\sigma_c)_{\text{calc}}$, equals $(\sigma_f V_f + \sigma_m' V_m)$, where σ_m' is the flow stress of the matrix at the yield strain of the fibres, and V_m is the volume fraction of the matrix; V_f is the volume fraction fibre loading [20]. The corresponding rule-of-mixtures for the Young's modulus is: $(E_c)_{\text{calc}} = (E_f V_f + E_m V_m)$. In this expression, E_f and E_m are the Young's modulus of the reinforcing fibres and the matrix, respectively. The ratios, $(\sigma_c)_{\text{calc}}/(\sigma_c)_{\text{meas}}$, or $(E_c)_{\text{calc}}/(E_c)_{\text{meas}}$, are shown in the uppermost portions of Figs. 5 through 8. In general, the ratios observed in this study do not differ significantly from unity; the ratio (grouped data) for the Young's modulus is 0.927 ± 0.062, whereas the ratio for the fracture stress is 1.020 ± 0.154.

Table IV shows a comparison of the mech-

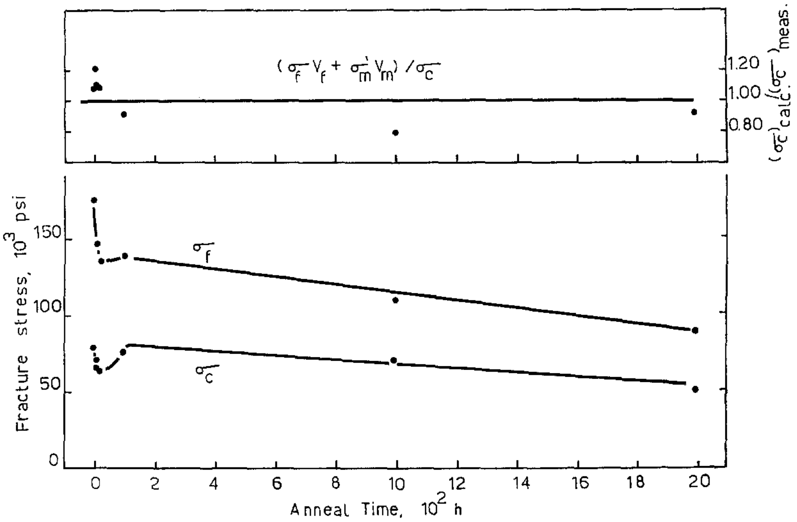

Figure 5 The effect of isothermal (925° C) annealing time on the fracture strength: composites (σ_c); extracted fibres (σ_f). The uppermost portion of the graph shows the ratio $(\sigma_c)_{calc}/(\sigma_c)_{meas}$.

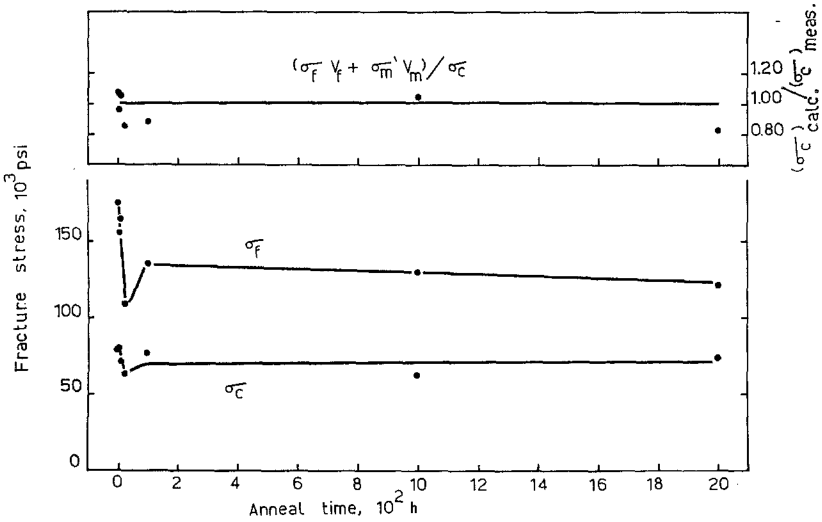

Figure 6 The effect of isothermal (1100° C) annealing time on the fracture strength: composites (σ_c); extracted fibres (σ_f).

anical properties of fibres extracted from composites annealed for 0.5 h in the temperature range 750 to 1200° C with the properties of stress-graphitized carbon fibres annealed tension-free in the absence of nickel. The mechanical properties of the extracted fibres are consistently lower than the corresponding properties of the relaxed fibres and the null-hypothesis probabilities indicate that the differences are significant. Therefore, although relaxation of the reinforcing fibres is a significant factor, the presence of nickel, either by itself or as an impurity source, is also a contributing factor in the mechanical property degradation of carbon-fibre, nickel-matrix composites. The contrasting results of Barclay and Bonfield [16] with those of Jackson and Marjoram [13] suggests that impurities may have a significant effect in weakening the fibres. However, the exact mechanism of *in situ* mechanical property degeneration was not successfully established in this study.

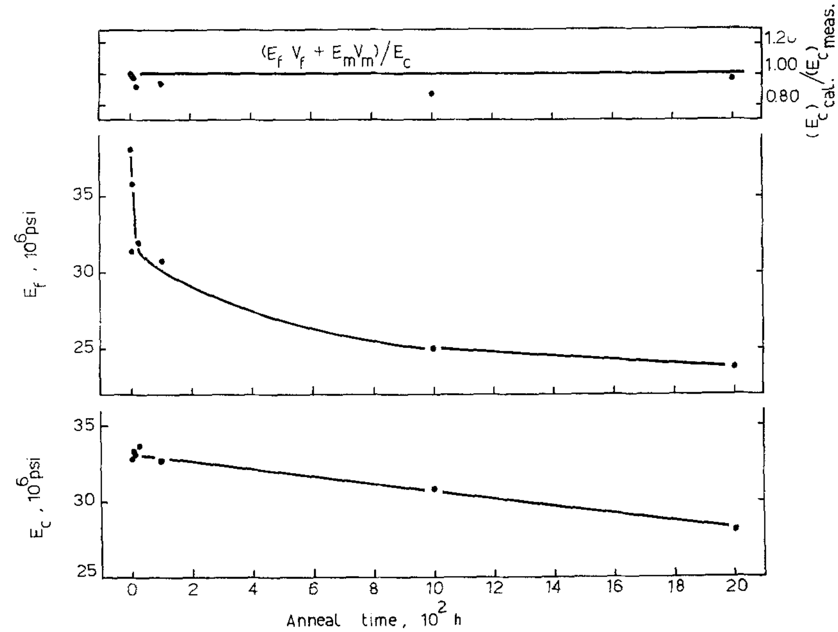

Figure 7 The effect of isothermal (925° C) annealing time on the Young's modulus: composites (E_c); extracted fibres (E_f). The uppermost portion of the graph shows the ratio $(E_c)_{calc}/(E_c)_{meas}$.

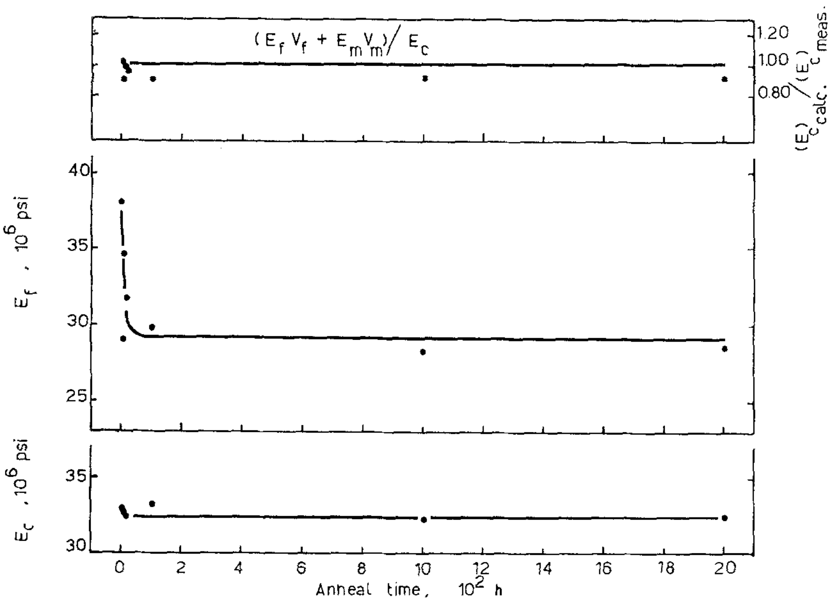

Figure 8 The effect of isothermal (1100°C) annealing time on the Young's modulus: composites (E_c); extracted-fibres (E_f).

4. Conclusions

Carbon-fibre, nickel-matrix composites are morphologically stable when the composites are annealed isothermally at temperatures less than 1200° C for periods of up to 200 h; fibre morphological degradation occurs when the composites are heated to temperatures in excess of 1250° C. The degradation temperature is lower

TABLE IV Comparison of the Young's modulus, E_f, and fracture strength, σ_f, of carbon fibres extracted from composites annealed for 0.5 h, and relaxed stress-graphitized carbon fibres [19].

		Temperature (°C)				
		750	925	1000	1100	1200
E_f, 10^6 psi	Relaxed fibres	47.0 ± 4.6	39.3 ± 5.8	40.0 ± 7.4	40.2 ± 6.4	42.7 ± 7.4
	Extracted fibres	34.5 ± 6.1	35.7 ± 5.3	25.6 ± 3.2	29.0 ± 8.3	20.2 ± 8.1
σ_f, 10^3 psi	Relaxed fibres	250 ± 75	221 ± 53	230 ± 60	211 ± 65	212 ± 41
	Extracted fibres	144 ± 44	175 ± 45	152 ± 52	155 ± 52	126 ± 41

(e.g. 1100°C) when the composites are exposed to thermal cycling or temperature gradients. Capillarity-related microstructural changes do not take place to any appreciable extent.

The fracture strength and Young's modulus of the reinforcing fibres decrease by 35 and 25% respectively when the nickel-coated filaments are hot-pressed into dense shapes; approximately half of this decrease can be directly attributed to relaxation of the ribbon-like fine structure of the fibres. The mechanical properties of the fibres are reduced further by about 20% when the hot-formed composites are re-annealed in the temperature range 750 to 1200°C. Most of this additional decrease occurs within 30 min from the start of the anneal; prolonged isothermal annealing does not result in further property degeneration. Impurities may have a causative relationship in producing fibre damage but the exact mechanism was not established in this study.

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